A Study of the Causes of a Stale Metallic Flavor in Strawberry Ice Cream

Together With Tests of Methods of Packing Berries

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A Study of the Causes of a Stale Metallic Flavor in Strawberry Ice Cream

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STRAWBERRY ICE CREAM is one of the most popular varieties of ice cream manufactured in the United States, representing 8.27 percent of the total ice-cream production in 1931.[4*] This flavor ordinarily is exceeded in sales only by vanilla and chocolate. Altho alcoholic extracts of strawberries are available for use in manufacturing ice cream, the entire berry is more commonly used because of the superior flavor imparted by the fruit constituents and the fact that consumers like to see the fruit in the ice cream.

Tho strawberries can be preserved in various ways, the frozen-pack method, because of its reduced cost and the resulting superior flavor of the berries, is being used by many ice-cream manufacturers. Investigators who have studied this method have reported results of variety tests, methods of packing, types of sugars used, storage temperatures, and in some cases flavor defects.[1-3,* 5-6,* 8-10*]

The main purpose of this study was to determine the cause of the stale metallic, or tallowy, flavor that is common in strawberry ice cream. Incidentally certain facts concerning the frozen-pack method of preserving berries were secured. The study extended over a period of four years (1930-1933) and covers so many different experimental runs that the method of procedure is best presented with the data.

SIX VARIETIES TESTED FOR USE IN ICE CREAM

Six common varieties of strawberries marketed in southern Illinois were tested to determine their relative merits as a source of flavor in ice cream. The average number of berries per quart, the average loss in stemming, and the rating on a flavor basis of the ice cream to which the fruit was added (15 percent by weight) are given in Table 1. Losses were due to the presence of stems, small, hard, and green berries, and overripe berries. As a rule the riper the fruit the greater

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*These numbers refer to literature citations on page 430.

was the shrinkage in stemming. The smaller berries often were of superior flavor but required more hours of labor in stemming.

The Dunlap berry, which produced the finest flavored ice cream, is not nearly so popular a market variety as the Aroma. This is probably due to the fact that altho the flavor and texture of the Dunlap is

TABLE 1.—COMPARISON OF STRAWBERRY VARIETIES FOR USE IN ICE CREAM

Varieties in order of flavor rating in ice cream	Average number berries per quart	Average loss in stemming
		perct.
1. Dunlap	101	10.5
2. Parson Beauty	280	7.5
3. Gandy	110	4.9
4. Premier	180	6.5
5. Gibson	102	5.5
6. Aroma	82	5.5

superior, the Aroma is a larger and more attractive berry and ships to better advantage. Parson Beauty was a very close second to the Dunlap in quality of flavor. The more highly flavored strawberry varieties such as the Dunlap, when available, should be used for flavoring ice cream.

The labor involved in preparing Dunlap berries for packing and the yield of packed strawberries is given in Table 2. These data are on the 1931 crop.

The average net weight of berries per crate was 24.5 pounds, the average shrinkage being 2.5 pounds.

TABLE 2.—PREPARATION OF DUNLAP STRAWBERRIES FOR STORAGE AT LOW TEMPERATURE

Shipment	Crates (24-quart)	Weight of berries	Man labor to stem and wash	Loss in stemming	Net yield[a]		
					2:1	3:1	4:1
		lbs.	*hrs.*	*perct.*	*gals.*	*gals.*	*gals.*
1	5	127	13	7.1	11	9	..
2	5	137	13.5	8.0	16	6	..
3	5	128	11.5	9.0	11	5	5
4	5[b]	117	11.2	8.5	8	4	4
5	5	141	12.5	8.8	22.5	..	..
Average per crate		27	2.5	2.5	4.20	3.85	3.70

[a]2:1 pack berries weighed 8.7 pounds per gallon; 3:1 pack berries weighed 8.50 pounds per gallon; 4:1 pack berries weighed 8.25 pounds per gallon. The proportions refer to parts of berries and sugar by weight.

[b]Less 16 quarts.

On the basis of the results obtained in these tests the approximate cost per gallon of berries ready for storage can be calculated by using the following formulas:

$$2{:}1 \text{ pack} = \frac{\text{cost of berries per crate} + (12.25 \times \text{cost of sugar per pound}) + 2.5 \times \text{cost of labor per hour}}{4.20}$$

$$3{:}1 \text{ pack} = \frac{\text{cost of berries per crate} + (8.17 \times \text{cost of sugar per pound}) + 2.5 \times \text{cost of labor per hour}}{3.85}$$

$$4{:}1 \text{ pack} = \frac{\text{cost of berries per crate} + (6.1 \times \text{cost of sugar per pound}) + 2.5 \times \text{cost of labor per hour}}{3.70}$$

For example, if the cost of berries is $2 a crate, sugar 5 cents a pound and labor 40 cents an hour, the cost per gallon of each of the three packs would be calculated as follows:

$$2{:}1 \text{ pack} = \frac{\$2 + (12.25 \times \$.05) + (2.5 \times \$.40)}{4.20} = \$.860$$

$$3{:}1 \text{ pack} = \frac{\$2 + (8.17 \times \$.05) + (2.5 \times \$.40)}{3.85} = \$.885$$

$$4{:}1 \text{ pack} = \frac{\$2 + (6.1 \times \$.05) + (2.5 \times \$.40)}{3.70} = \$.893$$

These cost figures do not represent total costs; such items as containers, refrigeration, and storage must be added. Variations from these figures will result with variations in amount of shrinkage and hours of labor.

PACKING BERRIES FOR STORAGE AT LOW TEMPERATURES

Addition of Sugar.—When strawberries are to be stored at low temperatures, the addition of sugar reduces the danger of fermentation before freezing and after thawing. The sugar also preserves the color and, to some extent, the natural flavor and aroma of the fruit, and keeps the berries from becoming too hard in the frozen ice cream. The addition of sugar, however, causes the fruit juice to be withdrawn

from the berry tissue by osmotic action. The greater the concentration of sugar on the outside of the berry the more complete will be the removal of fruit juice from the fruit tissue and the more shriveled will the fruit become. If, however, the berries are surrounded with an isotonic aqueous solution of cane or beet sugar, there will be little loss of juice by the fruit tissues and the fruit will retain its original shape.

Use of a Sugar Sirup.—In preserving peaches by freezing the use of a sugar sirup has many advantages over the use of sugar, according to Woodroof.[9*] His claims are as follows: (1) There is little or no change in fruit volume or in the juice volume when a sugar sirup is used. (2) The osmotic pressure of the sirup is near that of the peach juice. (3) The freezing point of the sirup is near that of the peach tissue. (4) The sirup is a better aid than sugar in preserving the color and texture on freezing and defrosting. (5) It is easier to get a uniform distribution and coverage of sirup than of sugar. (6) The use of prechilled sirup assures a more rapid cooling of the fruit than occurs in the sugar pack.

Many of these advantages in the use of a sirup in preserving peaches would very likely apply to the frozen-pack method of preserving strawberries.

Study of Different Methods.—In order to study the importance of the method of packing strawberries, three varieties—Howard 17, Burrill, and Dunlap—were packed without the addition of sugar, sirup, or water; with water; in three different concentrations of sirup; and with three different proportions of sugar (Table 3). After seven months of storage at 0° F., the berries were removed to room temperature and allowed to thaw gradually before being examined.

TABLE 3.—COMPARISON OF STRAWBERRIES FROZEN-PACKED IN VARIOUS WAYS

Pack	Flavor	Appearance	Color
Plain	Tasteless	Cell walls ruptured and collapsed	Lacking
Water	Tasteless	Slightly better than plain pack	Lacking
Sirup			
20 percent sirup	Somewhat tasteless	Nearly normal	Lacking
40 percent sirup	Fairly good	Normal	Good
60 percent sirup	Good	Shrunken and mushy	Good
Sugar			
2:1 sugar	Good	Similar to 60 percent sirup	Good
3:1 sugar	Good	Nearly normal	Good
4:1 sugar	Good	Nearly normal	Good

The larger proportions of sugar, whether added dry or in the form of a sirup, improved the palatability of the berries but detracted from their appearance when too great a concentration was used. If berries are to be used only in ice cream, their shape after storage is not of great importance. If, however, part of the pack is to be marketed for household use, such as for making strawberry cakes or pies, the melted berries should look as nearly normal as possible, in which case either the 3:1 or 4:1 sugar pack or the 40-percent sirup pack is recommended. Additional sugar may be needed either in the mix or in the berries when the light sugar pack (4:1) is used, if the finished ice cream is to be as sweet as is usually desired.

The best type and size of container to use for storing the berries depends upon the extent of the freezing operations in the plant. It has been the practice in the creamery at the University of Illinois to use gallon paper containers. This size of package is particularly convenient in small plant operations because the portions of the pack left unused are small. In large plants 50-gallon fir barrels paraffined on the inside may be practical. In smaller plants 30-gallon barrels or 10- and 5-gallon kegs may be more suitable, tho tinned containers, such as cream storage cans, are sometimes used.

COPPER SALT PROVES BASIC CAUSE OF METALLIC FLAVOR

The main object of this study, as previously stated, was to determine the cause of a peculiar off-flavor that sometimes develops in strawberry ice cream. The defect seems to be more common in winter than in summer, and some manufacturers are more troubled with the off-flavor than are others. The ice cream at the freezer has a normal flavor but upon storage, either in the hardening room or in the dealer's cabinet, changes sometimes occur that bring about a stale metallic-like flavor.

In preliminary studies attempts were made to ascertain whether the off-flavor might be caused by the variety of strawberries used, by the method of packing the berries, by aeration of the fruit, by yeast and mold contamination of the berries, by the presence of copper salts in the fruit, or by the source of the butterfat used in the mix.

Eight different varieties of Illinois grown berries packed in both sugar (4:1, 3:1, and 2:1 packs) and sirup (40 percent cane sugar) were used to flavor ice cream, but none of the experimental ice creams developed the flavor defect.

The effect of aeration was tested in a series of experiments in which ice cream[a] was used that was made with 15 percent strawberries that had been packed in 40 percent sugar sirup and aerated by permitting compressed air to bubble thru them for as long as 24 hours, a glass diffuser being used. While aeration of the fruit did cause a slight off-flavor in some of the ice creams, especially those containing

TABLE 4.—RELATION OF SOURCE OF BUTTERFAT TO THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN STRAWBERRY ICE CREAM

Description of sample	Flavor of ice cream after—	
	24 hours	2 weeks
94-score butter, no berries	Good	Good
94-score butter, 15 percent 2:1 frozen-pack berries	Good	Slight stale
89-score butter, no berries	High butter flavor	High butter flavor
89-score butter, 15 percent 2:1 frozen-pack berries	High butter flavor	Stale
88-score butter, no berries	Slight tallowy	Tallowy
88-score butter, 15 percent 2:1 frozen-pack berries	Strong metallic and tallowy	Very tallowy and stale

berries to which a copper-sulfate solution had been added, it did not seem to be of any great significance. The presence of the copper salt was of much greater importance than aeration.

To determine whether the growth of yeast and mold in the berries had any connection with the flavor defect, three lots of berries were inoculated with different cultures of pure yeast and three with different cultures of molds. After incubating the berries at room temperature for 24 hours, they were held at 40° F. for 24 hours and then added to ice cream at the rate of 15 percent. Duplicate sets were prepared, a small amount of a copper sulfate solution being added to one set. An uninoculated control incubated sample and one held at 40° F. were used in each test. After four months in storage the samples containing no copper were all of fairly good quality, none having the stale metallic flavor. The samples containing copper, on the other hand, were all off-flavor. The sample with the greatest degree of off-flavor was the one containing the uninoculated berries stored at 40° F.; the ice creams containing the berries inoculated with yeast had the most pronounced and most desirable strawberry flavor.

In a series of experiments in which the source of butterfat in the mix was varied, it was found that the stale metallic flavor was much

[a]Unless otherwise specified all experimental batches were frozen in a 40-quart direct expansion freezer.

more likely to develop in ice cream containing an 88- or 89-score butter than in that containing a 94-score butter (Table 4). The butter used in these mixes was melted and washed with water to remove the salt. The source of the serum solids was skim-milk powder. The mixes were pasteurized and homogenized after being standardized to contain 13 percent fat, 10.5 percent serum solids, 14 percent sugar, and .3 percent gelatin.

As a result of these preliminary tests it was concluded that a stale metallic-like flavor not unlike tallowiness will develop in ice cream containing a copper salt, and that the defect is greater when frozen-pack strawberries have been added to the ice cream. The fact that the flavor defect developed more rapidly in ice creams made from butter of relatively low score and in the presence of a copper salt suggested that the problem was one of butterfat oxidation.

STUDY OF FACTORS CONTRIBUTING TO DEVELOPMENT OF METALLIC FLAVOR

The stale metallic flavor having been identified as associated with the presence of a copper salt, the above study was continued in order to obtain more complete information on the nature of the reaction which takes place and the other contributing factors responsible for the flavor defect.

It was assumed that a certain amount of a copper salt had to be present in order to make possible the development of a stale metallic flavor. An experiment in which variable amounts of $CuSO_4$ were added showed that the off-flavor would develop when as much as one part of copper per million parts of mix was added, but the reaction was rather slow. In most of the trials about three parts of copper per million parts of mix were added, as this amount of copper usually produced the defect within a few days.

Commercial Packs Differed in Tendency to Cause Off-Flavor

To test possible differences in the readiness with which ice creams flavored with different brands of commercially packed berries developed an off-flavor, samples of strawberries preserved in various ways were obtained from several commercial dealers and added to ice cream at the rate of 8 percent by weight. The experimental mix used contained 13 percent fat, 10.5 percent serum solids, 14.5 percent sugar, .3 percent gelatin, and .25 percent dried whole egg, and was made from fresh milk products by the condensation process. Two sets of samples

were prepared, one set containing copper sulfate added at the rate of 3 parts of copper to a million parts of mix, while the second set contained no additional copper.[a] The ice creams were stored in an ice-cream hardening room in which the temperature usually fluctuated between 0 and –10° F. The flavor of samples after storage for 3, 10, and 40 days in the hardening room is given in Table 5.

TABLE 5.—DEVELOPMENT OF OFF-FLAVOR IN ICE CREAM FLAVORED WITH DIFFERENT BRANDS OF STRAWBERRIES

Method of packing berries	Copper added[a]	Flavor of ice cream after—		
		3 days	10 days	40 days
1. Control—no berries	None	Good	Good	Good
Control—no berries	$CuSO_4$ (3 p.p.m.)	Good	Good	Slight stale metallic
2. Processed[b]	None	Good	Good	Good
Processed[b]	$CuSO_4$ (3 p.p.m.)	Good	Good	Slight stale
3. Processed[b]	None	Good	Good	Good
Processed[b]	$CuSO_4$ (3 p.p.m.)	Slight stale metallic	Slight stale metallic	Stale metallic
4. Processed[b]	None	Good	Good	Good
Processed[b]	$CuSO_4$ (3 p.p.m.)	Slight stale metallic	Slight stale metallic	Slight stale metallic
5. Cold pack	None	Good	Good	Good
Cold pack	$CuSO_4$ (3 p.p.m.)	Strong stale metallic	Very strong stale metallic	Very strong stale metallic
6. Vacuum cold pack	None	Good	Good	Good
Vacuum cold pack	$CuSO_4$ (3 p.p.m.)	Strong stale metallic	Strong stale metallic	Strong stale metallic
7. Cold pack	None	Good	Good	Good
Cold pack	$CuSO_4$ (3 p.p.m.)	Slight stale metallic	Strong stale metallic	Very strong stale metallic
8. Cold pack	None	Good	Good	Good
Cold pack	$CuSO_4$ (3 p.p.m.)	Stale metallic	Strong stale metallic	Very strong stale metallic
9. Cold pack	None	Good	Good	Good
Cold pack	$CuSO_4$ (3 p.p.m.)	Good	Slight stale metallic	Stale metallic

[a]In all tests parts per million refers to parts of copper per million parts of mix.
[b]Heated when canned but exact heat treatment not reported by manufacturer.

When the mix was not contaminated with copper, there was no trouble from a stale metallic flavor in the strawberry ice cream made with any of the brands of berries used. All were rated as good at the

[a]In all tests either the entire mix or the condensed milk was prepared in an Allegheny metal vacuum pan. This type of metal has been shown to have no effect upon the flavor of dairy products.

end of the test periods. The heated packs—Nos. 2, 3, and 4—had less tendency to bring about the flavor than did the unheated packs. In the plain ice cream only the 40-day sample contaminated with copper developed a stale metallic flavor and then the flavor was only slight.

Thus it would appear that strawberries may contain a factor that has the power to catalyze the reaction responsible for the off-flavor described as stale metallic, and that heated packs have less tendency than unheated packs to cause the flavor.

Heating of Fruit Retarded Off-Flavor

Several experiments were performed to determine to what extent the heat treatment given the berries was a factor in preventing the off-flavor (Table 6).

None of the ice-cream samples in which heated berries were used acquired the off-flavor as quickly as did that in which the raw fruit was

TABLE 6.—EFFECT OF HEATING STRAWBERRIES ON THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN ICE CREAM

Treatment of berries	Extent of stale metallic flavor[a] developed after—		
	12 days	18 days	77 days
Control—unheated	±	+	++++
Autoclaved at 15 pounds pressure for 15 minutes	None	±	+
Held at 150° F. for 30 minutes	None	±	++++
Held at 175° F. for 30 minutes	None	±	+++
Held at 200° F. for 30 minutes	None	±	++

[a] ± indicates that the presence of the flavor defect is questionable; + indicates the degree of off-flavor.

used. Heating the berries to 175° F. or higher was more effective in retarding the development of the off-flavor in the ice cream than heating them to 150° F. Heating did not, however, prevent the defect, for all samples developed a stale metallic flavor after storage for 77 days in the hardening room.

The ice-cream mix used in this experiment was made from 40-percent cream, skim milk, and concentrated skim milk. Copper contamination, such as often occurs commercially, was introduced by pasteurizing the mix in a tinned copper steam kettle on which much of the tin was worn away, exposing the copper. The berries used were of the Dunlap variety; they had been stored with sugar at hardening

room temperature for ten months. Ten percent of fruit was added in each test.

Amount of Fruit Added Is Important

Some of the same mix described in the preceding experiment was flavored with varying amounts of both raw and heated strawberries as indicated in Table 7. Within the period of the experiment (77 days)

TABLE 7.—RELATION BETWEEN AMOUNT OF STRAWBERRIES ADDED TO ICE CREAM AND THE DEVELOPMENT OF A STALE METALLIC FLAVOR

Proportion of berries	Extent of stale metallic flavor developed after—		
	12 days	17 days	77 days
Control—no berries	None	None	None
Unheated			
1 percent	+++	+++	++++++
5 percent	++	+++	+++++
10 percent	±	+	++++
15 percent	None	None	None
25 percent	None	None	None
50 percent	None	None	None
Autoclaved[a]			
1 percent	+	++	+++
5 percent	±	+	++
10 percent	None	±	+
15 percent	None	None	None
25 percent	None	None	None
50 percent	None	None	None

[a]Heated in an autoclave for 15 minutes at 15 pounds pressure.

none of the samples containing 15 percent or more fruit acquired an off-flavor, whereas all the lots containing 10 percent or less fruit had a stale metallic flavor at the end of 17 days. The most pronounced off-flavor was in those samples containing the unheated berries.

Juice of Berry Contains Factor Causing Off-Flavor

To determine what portion of the strawberry was responsible for the development of the stale metallic flavor in strawberry ice cream, the following experiment was performed.

Frozen-pack Dunlap berries were used. The mix contained 13 percent fat, 10.5 percent serum solids, 14 percent sugar, .25 percent whole egg yolk, and .3 percent gelatin. The milk products in this mix were 40-percent cream, sweetened condensed skim milk, and skim milk. Seven different lots of ice cream were prepared, six containing 10 percent of a certain portion of the berry, as follows: (1) whole berries; (2) washed berry fiber; (3) unwashed berry fiber; (4) berry tissue and juice that passed thru cheese cloth and contained no seeds;

(5) very fine tissue and juice caught on filter paper, containing no seeds; (6) juice filtered thru quantitative filter paper. The control lot contained no berries. All the mixes contained 2.6 parts per million of copper added as $CuSO_4$.

After one day in storage all lots had good flavor; after 10 days in storage the lots containing the whole fruit, tissue and juice, very fine tissue and juice, and filtered juice had developed a stale metallic flavor (Table 8). After 43 days in storage the stale metallic flavor was still

TABLE 8.—RELATION OF DIFFERENT BERRY FRACTIONS TO THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN ICE CREAM

Berry fraction—added to mix at rate of 10 percent by weight	Flavor of ice cream after—		
	1 day	10 days	43 days
Control—no berries	Good	Doubtful	Slight stale metallic
Whole fruit	Good	Stale metallic	Stale metallic
Washed fiber	Good	Good	Fair
Unwashed fiber	Good	Good	Fair
Tissue and juice	Good	Stale metallic	Stale metallic
Very fine tissue and juice	Good	Stale metallic	Stale metallic
Filtered juice	Good	Stale metallic	Stale metallic

evident in these samples and in addition a slight stale metallic flavor was present in the control lot. The samples containing washed and unwashed fiber lost their fresh flavor after 43 days but did not develop the characteristic stale metallic flavor of the other samples.

Strawberries apparently contain two factors related to the development of the flavor defect. One is associated with the fiber and aids in preventing the reaction responsible for the off-flavor; the other agent is contained in the juice and tissue mixture and has the effect of hastening the reaction.

Flavor Defect Associated With Presence of Butterfat

To determine the role that butterfat might play in the development of the off-flavor in strawberry ice cream, the following experiment was performed. Using fresh 40-percent cream and sweetened condensed skim milk, mixes were prepared containing different proportions of fat and total solids, as indicated in Table 9.

The mixes contained no gelatin and were processed at low pressure (about 200 pounds) thru a small emulsifier. Ice creams were made with each of the mixes as follows, and frozen in a hand freezer: (a) control—no additional treatment; (b) $CuSO_4$ added at the rate of 4 p.p.m.

TABLE 9.—RELATION OF BUTTERFAT TO THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN STRAWBERRY ICE CREAM

Description of sample[a]	Flavor of ice cream after—	
	4 days	40 days
.144 percent fat, 24.96 percent total solids		
Control—no berries	Good	Good
No berries—$CuSO_4$ (4 p.p.m.)	Good	Unclean
10 percent frozen-pack	Good	Good
10 percent frozen-pack plus $CuSO_4$ (4 p.p.m.)	Good	Very unclean
4.55 percent fat, 31.44 percent total solids		
Control—no berries	Good	Good
No berries—$CuSO_4$ (4 p.p.m.)	Stale metallic ++	Stale metallic +++
10 percent frozen-pack	Good	Good
10 percent frozen-pack plus $CuSO_4$ (4 p.p.m.)	Stale metallic +	Stale metallic ++++
8.98 percent fat, 35.76 percent total solids		
Control—no berries	Good	Good
No berries—$CuSO_4$ (4 p.p.m.)	Stale metallic +++	Stale metallic +++
10 percent frozen-pack	Good	Good
10 percent frozen-pack plus $CuSO_4$ (4 p.p.m.)	Stale metallic ++	Stale metallic +++++++

[a]In all tests parts per million refers to parts of copper per million parts of mix.

of copper; (c) 10 percent frozen-pack strawberries used; (d) same as c plus $CuSO_4$ at the rate of 4 p.p.m. of copper. Criticisms on the samples are given in Table 9.

Altho an unclean flavor developed in the mixes containing .144 percent fat, to which copper had been added, the off-flavor was not nearly so pronounced nor so pungent as that occurring in corresponding samples containing 4.55 percent fat. In the same way, the off-flavor in the samples containing 4.55 percent fat, to which copper had been added, was not so intense as that in the corresponding samples containing 8.98 percent fat. Thus the flavor defect was quite evidently associated with the presence of butterfat.

It would have been of interest to study the development of the off-flavor in a fat-free ice cream. The presence of fat in all skim milk products would, however, make it impossible to build a mix from condensed or fluid skim milk without there being enough fat present to develop a small amount of the off-flavor.

Citric Acid Content of Strawberries Is Not Important

Inasmuch as strawberries contain fruit acids, such as citric, the effect of variations in the citric-acid content of strawberry ice cream

upon the development of the stale metallic flavor was studied. A mix containing 10 percent fat, 10 percent serum solids, and 15 percent sugar was prepared from 40-percent cream and sweetened condensed skim milk and processed with a small emulsifier at approximately 200 pounds pressure. Instead of using the whole strawberry the filtered juice was used, since this fraction of the berry had been found to contain the substance responsible for the off-flavor in strawberry ice cream. A portion of the juice was reduced in acidity by the addition of $NaHCO_3$ before it was added to the ice cream.

The following pH determinations were made: raw berry juice, 3.37; neutralized juice, 6.00; mix-control, 6.43; mix plus $CuSO_4$ (3 p.p.m.), 6.47; mix plus raw juice plus $CuSO_4$ (3 p.p.m.), 6.27.

TABLE 10.—EFFECT OF VARYING THE CITRIC ACID CONTENT OF STRAWBERRIES ON THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN ICE CREAM

Description of sample[a]	Flavor of ice cream after—	
	4 days	28 days
1. Control—no berries	Good	Doubtful
2. 10 percent raw juice	Good	Doubtful
3. No berries—$CuSO_4$ (3 p.p.m.)	Stale metallic +++	Stale metallic ++++++
4. 10 percent raw juice, $CuSO_4$ (3 p.p.m.)	Stale metallic ++	Stale metallic +++++++
5. 10 percent neutralized juice, $CuSO_4$ (3 p.p.m.)	Good	Stale metallic ++++++
6. Same as No. 5, plus .17 percent citric acid	Good	Stale metallic +++++
7. Same as No. 3, plus .17 percent citric acid	Good	Stale metallic +++++
8. 10 percent raw juice, $CuSO_4$ (3 p.p.m.), .17 percent citric acid	Good	Stale metallic +++++
9. Same as No. 7, except .34 percent citric acid	Good	Stale metallic ++++
10. 10 percent raw juice, $CuSO_4$ (3 p.p.m.), .34 percent citric acid	Good	Stale metallic ++++
11. Same as No. 7, except .68 percent citric acid	Good	Stale metallic +++
12. 10 percent raw juice, $CuSO_4$ (3 p.p.m.), .68 percent citric acid	Good	Stale metallic +++

[a]In all tests parts per million refers to parts of copper per million parts of mix.

The flavor criticisms of the ice cream are given in Table 10. Neither the addition of sodium bicarbonate nor of citric acid to the strawberry juice before the juice was added to the ice cream containing $CuSO_4$ (3 p.p.m.) caused an increase in the stale metallic flavor. As a matter of fact, the flavor seemed less pronounced in the samples to which the acid was added. This precludes the possibility of the citric-acid content of strawberries being responsible for catalyzing the reaction producing the stale metallic flavor. As indicated by the pH of the mix before and after 10 percent raw juice was added, the buffer

action of the mix constituents is apparently great enough to offset practically all effect which the free hydrogen ions in the fruit juice may have upon the pH concentration of the flavored mix.

Homogenization of Mix Retarded Off-Flavor

In a previous study[7]* of the tallowy flavor in pasteurized milk, the authors found that homogenized milk contaminated with copper did not acquire so tallowy a flavor when stored at 40° F. as did the same milk unhomogenized. In the present study it was likewise found that strawberry ice cream made from a homogenized mix was less likely to develop the stale metallic flavor than one made from an unhomogenized mix (Table 11).

The mix used contained 40-percent cream, skim milk, and 32-percent concentrated skim milk as a source of milk solids, 12 percent fat,

TABLE 11.—EFFECT OF HOMOGENIZATION OF MIX ON THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN STRAWBERRY ICE CREAM

Treatment of sample	Flavor of ice cream after 4 days
Unhomogenized	
No berries	Metallic
10 percent berries	Stale metallic (strongest off-flavor of all)
Homogenized	
1,000 pounds on 1st valve	
No berries	Good
10 percent berries	Stale metallic
2,500 pounds on 1st valve; 1,000 pounds on 2d valve	
No berries	Good
10 percent berries	Very slight metallic
3,500 pounds on 1st valve	
No berries	Good
10 percent berries	Very slight metallic

10.5 percent serum solids, 14 percent sugar, and .3 percent gelatin. $CuSO_4$ was added at the rate of 3 parts of copper per million parts of mix.

After being pasteurized in the usual way by being heated to 145° F. and held at that temperature for 30 minutes, samples of the mix were pumped thru a homogenizer at different pressures; namely: at 1,000 pounds pressure on the first valve, at 2,500 pounds pressure on the first valve and 1,000 pounds on the second, and at 3,500 pounds pressure on

the first valve. Samples homogenized at 1,000 pounds pressure had a more pronounced off-flavor than those homogenized at the higher pressures.

The freezing was done in hand freezers and the berries were added at the rate of 10 percent by weight. Frozen-pack Gibson strawberries were used.

Other Fruits Than Strawberries May Cause Off-Flavor

To study the effect of fruits other than strawberries on the development of the stale metallic flavor in ice cream, a batch of ice cream containing 13 percent fat, 10 percent serum solids, 14 percent sugar, and .3 percent gelatin was divided into small lots which were flavored with 3, 10, and 25 percent each of different fruits and vegetables.

The stale metallic flavor developed in the ice creams containing 3 and 10 percent of oranges, lemons, pineapple, apples, potatoes, peaches, and apricots, respectively. The ice creams flavored with rhubarb and tomatoes did not develop the defect. None of the ice cream containing 25 percent of the above flavoring materials developed an off-flavor.

OXIDATION THEORY TESTED BY OXIDATION-REDUCTION POTENTIAL MEASUREMENTS

Eh Measurements Show Off-Flavor Is Due to Fat Oxidation

Evidence already presented suggests that fat oxidation in strawberry ice cream was the cause of the stale metallic flavor. It was shown that a copper salt must be added, either to the fruit or to the mix, in order to produce the off-flavor and that when the butterfat in strawberry ice cream came from an 88-score butter, the off-flavor developed, but when a 94-score butter was used there was no suggestion of the stale metallic flavor. In another experiment it was found that a certain amount of butterfat must be present in the ice cream in order to develop a distinct stale metallic flavor.

To secure further evidence that the stale metallic flavor is nothing more than a modified tallowiness, oxidation-reduction potential measurements were made in a series of experiments designed to introduce some of the variables already studied. This method was used by the authors in the study of tallowiness in market milk previously referred to.[7]*

The test is based on the fact that as the intensity of oxidation is increased, the voltage exerted on a platinum electrode is also increased,

and vice versa. The E.M.F. was measured with a Leeds and Northrup Type K potentiometer and a sensitive galvanometer. Connections were made from the saturated KCl calomel half-cell, as the reference electrode, to the samples under measurement by means of a saturated KCl liquid junction and saturated KCl agar bridges. Potential readings were reduced to the conventional hydrogen scale.

A mix containing 12 percent fat, 10.5 percent serum solids, 14 percent sugar, and .33 percent gelatin was used. This mix was made from 40-percent cream, skim milk, and 32-percent concentrated skim milk. Frozen-pack Gibson berries were used. The fruit was added to the mix before freezing in hand freezers. The following mixes were prepared: (1) control mix, no berries added; (2) 10 percent berries added; (3) no berries, $CuSO_4$ added at rate of 1 p.p.m.; (4) 10 percent berries and $CuSO_4$ (1 p.p.m.) added; (5) no berries, $CuSO_4$ added at rate of 3 p.p.m.; (6) 10 percent berries and $CuSO_4$ (3 p.p.m.) added.

It will be observed that the addition of the berries alone to the ice cream caused the potential to shift towards the side of reduction, but that the potential reading remained practically constant during the course of the experiment (Table 12). However, when a copper salt

TABLE 12.—RELATION BETWEEN OXIDATION-REDUCTION POTENTIAL READINGS AND THE DEVELOPMENT OF A STALE METALLIC FLAVOR IN STRAWBERRY ICE CREAM

Description of sample[a]	Eh when made	Eh after 1 day	Eh after 3 days	Flavor after 3 days	Eh after 33 days	Flavor after 33 days
Control—no berries	.50452	.49687	.50242	Good	.50493	Good
10 percent berries	.33693	.34623	.34893	Good	.34451	Good
No berries—$CuSO_4$ (1 p.p.m.)	.49826	.50893	.51212	Good	.50033	Slight unclean
10 percent berries, $CuSO_4$ (1 p.p.m.)	.36382	.37873	.38806	Doubtful	.45346	Strong stale metallic
No berries—$CuSO_4$ (3 p.p.m.)	.48823	.49957	.50757	Doubtful	.50093	Slight unclean
10 percent berries, $CuSO_4$ (3 p.p.m.)	.41182	.46357	.48097	Strong stale metallic	.48403	Very strong stale metallic

[a]In all tests parts per million refers to parts of copper per million parts of mix.

was added along with the berries, the potential did not drop so far towards the side of reduction even at the beginning of the experiment, and during the storage period the potential apparently gradually changed toward the side of oxidation. Furthermore, as the potential of

these samples shifted towards oxidation, the flavors of the ice cream also changed from normal to the characteristic stale metallic flavor, indicating a correlation between the oxidation reaction and the development of the flavor defect. There was but little change in potential towards reduction in those samples to which copper was added but which contained no strawberries. This bears out the contention that the berries contain a factor which, in the presence of copper, catalyzes the oxidation reaction. Undoubtedly in time the potential of the unflavored ice cream containing copper would have shifted towards oxidation and a tallowy flavor would have become evident.

Different Amounts and Different Parts of Berries Cause Varying Degrees of Oxidation

The stale metallic flavor having been shown by organoleptic methods to have developed faster with small additions of fruit (less than 10 percent) and to have become more pronounced than when larger amounts of fruit (over 10 percent) were added, it was desired to study the oxidation-reduction potential in such samples. Accordingly the following experiment was conducted.

TABLE 13.—EFFECT OF INCREASING AMOUNTS OF STRAWBERRIES ON THE OXIDATION-REDUCTION POTENTIAL OF THE ICE-CREAM MIX

Proportion of berries	Eh after aging the mix at 40° F. for 24 hours
Control—no berries	.43367
5 percent	.39764
10 percent	.39977
20 percent	.33244
30 percent	.30139

The mix used contained 12 percent fat, 10 percent serum solids, 14 percent sugar, and .3 percent gelatin, and was prepared by the condensation process, using 40-percent cream and skim milk as the source of the milk solids. Samples containing 5, 10, 20, and 30 percent frozen-pack Dunlap berries were tested. The potential readings were made 24 hours after the samples had been flavored and stored at 40° F.

An increased amount of strawberries added to the ice-cream mix tended to cause a reduced Eh reading (Table 13). The strong reducing powers of strawberries cannot be explained entirely thru dilution, however, since the berries alone gave an Eh reading of .28440. Appar-

ently the addition of a large percentage of berries sets up a system that is capable of resisting oxidation.

Experiments were also made to determine the effect of different portions of frozen-pack berries on the oxidation-reduction potential of the ice-cream mix. The mix was similar and the berries were the same as those used above. The berry fractions (same as described on page 420) were added before aging for 24 hours at the rate of 10 percent by weight.

The low Eh of the mix containing the washed and unwashed fiber indicated that it is partly this constituent of the berry that causes the drop in Eh when the fruit is added to the ice cream (Table 14). The

TABLE 14.—EFFECT OF DIFFERENT STRAWBERRY FRACTIONS ON THE OXIDATION-REDUCTION POTENTIAL OF THE ICE-CREAM MIX

Berry fraction—added to mix at rate of 10 percent by weight	Eh after aging the mix at 40° F. for 24 hours
Control—no berries	.44970
Whole fruit	.40193
Washed fiber	.33485
Unwashed fiber	.33781
Tissue and juice	.38218
Very fine tissue and juice	.39361
Filtered juice	.36182

TABLE 15.—EFFECT OF ADDING STRAWBERRIES TO MIX 24 HOURS BEFORE FREEZING ON THE OXIDATION-REDUCTION POTENTIAL

Description of sample[a]	Eh of mix 2 hours after freezing	Eh of melted ice cream 24 hours after freezing	Flavor of ice cream 21 days after freezing
10 percent berries at freezer	.46469	.43396	Good
10 percent berries at freezer, $CuSO_4$ (3 p.p.m.)	.46630	.45056	Stale metallic
10 percent berries to mix 24 hours before freezing, $CuSO_4$ (3 p.p.m.)	.28200	.20037	Slight puckery

[a]In all tests parts per million refers to parts of copper per million parts of mix.

juice passed thru a filter paper appeared to have similar reducing powers.

When the juice was passed thru a Pasteur-Chamberlain filter, however, and then added to the mix, a higher Eh resulted. The Eh of the mix to which no juice was added was .43536; with 10 percent juice

filtered thru the paper it was .29907; with 10 percent juice filtered thru a Pasteur-Chamberlain, .40566. Apparently there was a reducing substance contained in the juice which was mostly filterable.

Adding the fruit to the mix a few hours before the mix is frozen enables the factor contained in the berries that is responsible for the reducing effect to function to better advantage, as is indicated by the data in Table 15. The mix used in this test was made from 14-percent cream and contained 15 percent sugar, 1 percent fresh eggs, but no gelatin.

SUMMARY AND CONCLUSIONS

The elimination of copper contamination is the most necessary step in preventing the development of a stale metallic flavor in strawberry ice cream. This off-flavor is associated with the oxidation of the butterfat, as shown by oxidation-reduction measurements made on experimental batches of ice cream. The addition of copper to the ice cream mix catalyzes the oxidation reaction. Ice-cream plant operators need not be troubled with this flavor defect if they exercise proper care in the selection of dairy products and take the necessary precautions to avoid copper contamination of the mix or any of its constituents.

The development of a stale metallic flavor in strawberry ice cream can be retarded by homogenizing the mix at a high pressure, by heating the berries, by soaking the fruit in the mix before freezing, or by increasing the amount of the fruit added or increasing the fiber content of the berries.

Since the off-flavor developed more rapidly in ice-cream mixes to which strawberries were added than in the control mix to which no berries were added, and since the use of the fruit fiber was shown to check the development of the defect, strawberries apparently contain two agents affecting fat oxidation, one serving as a catalyst and the other as a reducing agent. The former is contained in the juice and the latter is associated with the fibrous material. Increasing the citric acid content of the berries did not hasten the development of the flavor defect.

The fact that different commercial pack berries varied in their ability to cause the stale metallic flavor can probably be attributed to the differences in concentration of pack or to the proportion of fiber as well as to the heat treatment given the berries before or after canning. Fruits other than strawberries, such as oranges, lemons, and pineapple, were also found to accelerate the reaction responsible for the off-flavor.

Six of the most common varieties of strawberries grown in southern Illinois were tested for their desirability as a source of flavor in ice cream. These varieties ranked in the following order of preference: Dunlap, Parson Beauty, Gandy, Premier, Gibson, Aroma. The average net weight of Dunlap berries was found to be 24.5 pounds per case of 24 boxes. The average yield per case of 2:1 pack was 4.2 gallons; of 3:1 pack, 3.85 gallons; and 4:1 pack, 3.7 gallons. Either 2, 3, or 4 parts of berries to 1 part of sugar made a desirable pack so far as flavor was concerned. When it was desired to keep the fruit whole, a pack of 3 or 4 parts of berries to 1 part of sugar or 40 percent sugar sirup solution was found to be preferable.

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